

TITLE PAGE

SOP NO. HW-29

Revision 1

October 2001

STANDARD OPERATING PROCEDURE FOR THE VALIDATION OF ORGANIC DATA

ACQUIRED USING METHOD 524.2 (Revision 4.1, 1995)

MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(GC/MS) :CAPILLARY COLUMN

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA Method 524.2. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA Method 524.2 and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", October 1999 (EPA - 540/R-99-008). This document covers technical as well as method specific problems; however situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 24.

The reviewer must prepare a detailed data assessment to be submitted along with the complete SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data, and contract non-compliance.

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YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ LAB: _____

SITE NAME: _____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? ☐ ☐ ☐

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, signed release, or cover letter present? ☐ ☐ ☐

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? ☐ ☐ ☐

II. VOLATILE ANALYSES**1.0 Traffic Reports and Laboratory Narrative**

1.1 Are the Traffic Reports, Chain of Custodies, or signed releases from the field samplers present for all samples? ☐ ☐ ☐

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? ☐ ☐ ☐

1.3 Sample Conditions/Problems

1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ☐ ☐ ☐

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YES NO N/A

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated ($>10^{\circ}\text{C}$), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of _____ [] _____ collection to date of analysis, been exceeded?

The holding time for aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with acid to a pH <2 and stored at 4°C , then aqueous samples must be analyzed within 14 days from time of collection. If uncertain about preservation, contact the laboratory/sampling team to determine whether or not samples were preserved.

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms ? [] _____

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms ? [] _____

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YES NO N/A

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data Assessments and contact the laboratory/project officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were outliers marked correctly with an asterisk? ☐ ☐ ☐

ACTION: Circle all outliers with a red pencil.

3.4 Were one or more volatile surrogate recoveries outside required limits for any sample or method blank (Surrogate recovery is 70-130% for aqueous samples) ☐ ☐ ☐

NOTE: Lab can use their developed in house acceptance criteria, (See Method 8000B Sect.8.7) if none, then use 70-130%.

If yes, were samples reanalyzed? ☐ ☐ ☐

Were method blanks reanalyzed? ☐ ☐ ☐

ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and reported data? ☐ ☐ ☐

ACTION: If large errors exist, take action as specified in section 3.2 above.

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YES NO N/A

4.0 Laboratory Fortified Blanks (CLP Form III Equivalent)

- 4.1 Have the volatile Laboratory Fortified Blanks (LFB) recoveries been listed on the laboratory reporting form? ☒ ☐ ☐

NOTE: If the data has not been reported, then contact the laboratory/project officer to obtain the information necessary to evaluate the spike recoveries in the MS, MSD, and LFB. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

NOTE: The LFB spike is spiked with the same analytes at the same concentrations as a calibration standard (Method 524.2-16, Sect.9.3) if different, make note in Data Assessment.

- 4.2 Were Laboratory Fortified Blanks analyzed at the required frequency (1 LFB per 20 samples)? ☒ ☐ ☐

ACTION: If any LFB data are missing, take the action specified in section 3.2 above.

- 4.3 How many LFB volatile spike recoveries are outside QC Limits?

Water out of

ACTION: Circle all outliers with a red pencil.

- 4.4 Were one or more of the volatile LFB recoveries outside 70-130% recovery as per Method 524.2-17, Sect.9.6 ☐ ☒ ☐

- ACTION:
1. If the recovery is > upper in-house limit (or 130%), only positive values for the affected analytes of the compound(s) are flagged "J".
 2. If the recovery is < lower in-house limit (or 70%), flag positive values for the affected analytes of the compound(s) "J" and non detects "J".

NOTE: All analytes in associated sample results are qualified

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YES NO N/A

for the following criteria:

1. If 25% of the LFB recoveries were < lower in-house limit (or 70%) qualify all positive results "J" and all non-detects "R".
2. If two or more LFB recoveries were < 10% qualify all positive results "J" and all non-detects "R".

5.0 Laboratory Fortified Sample Matrix (LFM)

NOTE: Analysis of a laboratory fortified sample matrix (LFM) is required ONLY if the criteria in section 9.4 are not met. "The integrated areas of the quantitation ions of the internal standards and surrogate in all samples, continuing calibration checks and blanks should remain reasonably constant over time". An abrupt change may indicate a matrix effect and a laboratory fortified duplicate sample must be analyzed to test for matrix effect.

5.1 Have the volatile Laboratory Fortified Sample Matrix (LFM) recoveries been listed on the laboratory reporting form? ☐ ☐ ☐

NOTE: The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

5.2 Were Laboratory Fortified Sample Matrix (LFM) analyzed at the required frequency? ☐ ☐ ☐

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a Laboratory Fortified Duplicate Sample (LFM) should be analyzed (Method 524.2-17, Sect.9.4)

ACTION: No action is taken on LFM data alone. However using professional judgement, the validator may use the LFM results in conjunction with other QC criteria and qualify data for that matrix following the guidelines addressed in Sections 4.3 to 4.4.

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	YES	NO	N/A
6.0 <u>Laboratory Reagent Blanks (LRB)</u>			
6.1 Is the LRB Summary form present? _____	<input type="checkbox"/>	_____	_____
6.2 Frequency of Analysis: Has a Laboratory reagent blank been reported for samples of similar matrix, or concentration level, and for each extraction batch?	<input type="checkbox"/>	_____	_____
6.3 Has a LRB been analyzed for each GC/MS system used ?	<input type="checkbox"/>	_____	_____
ACTION: If any LRB data are missing, take action as specified in section 3.2. If not available, use professional judgement to determine if the associated sample data should be qualified.			
6.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for the volatiles?	<input type="checkbox"/>	_____	_____
ACTION: Use professional judgement to determine the effect on the data.			
7.0 <u>Contamination</u>			
7.1 Are there field reagent blanks (FRB) associated with every sample?	<input type="checkbox"/>	_____	_____
ACTION: If no, note in Data Assessment that there is no associated field reagent blank. For analytes with high concentrations, use professional judgement on qualification of these values and make note in Data Assessment. Duplicate FRB's must be handled along with each sample set, which is composed of the samples collected from the same general site at approximately the same time.			
7.2 Do any Laboratory reagent blank/Field reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor.	_____	<input type="checkbox"/>	_____
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (May attach a separate sheet.)			
NOTE: All field reagent blank results associated with a			

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particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field reagent blanks/ Laboratory reagent blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank value	Sample conc < CRQL & <10x blank value	Sample conc > CRQL & >10x blank
Methylene Chloride Acetone Toluene 2-Butanone	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank
Other contam- inants	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

NOTE: The reporting of TIC compounds may or may not be required.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" unusable.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) ☒ ☐ ☐ for analysis of volatiles by Method 524.2? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL (Supelco) or equivalent column. (Method 524.2-9, Sect. 6.3.2)

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YES NO N/A

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed? ☐ ☐ ☐

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? ☐ ☐ ☐

9.3 Has an instrument performance check solution (BFB) been analyzed for every twelve hours of sample analysis per instrument?(Method 524.2-18, Sect. 10.1) ☐ ☐ ☐

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If the laboratory/project officer/appropriate official cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, ("R").

9.4 Have the ion abundances been normalized to m/z 95? ☐ ☐ ☐

9.5 Have the ion abundance criteria been met for each instrument used? ☐ ☐ ☐

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values) ☐ ☐ ☐

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YES NO N/A

but if errors are found, check more.)

- 9.7 Have the appropriate number of significant Figures (two) been reported? ☐ ☐ ☐

ACTION: If large errors exist, take action as specified in section 3.2.

- 9.8 Are the spectra of the mass calibration compound acceptable? ☐ ☐ ☐

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

10.0 Target Analytes (CLP Form I Equivalent)

- 10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| a. Samples and/or fractions as appropriate | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| b. Laboratory Fortified Sample Matrix | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| c. Blanks | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| d. Laboratory Fortified Blank | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

- 10.2 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| a. Samples and/or fractions as appropriate | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| b. Laboratory Fortified Sample Matrix
(Mass spectra not required) | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| c. Blanks | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| d. Laboratory Fortified Blanks | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

ACTION: If any data are missing, take action specified in 3.2 above.

- 10.3 Is chromatographic performance acceptable with respect to:

- | | | | |
|---------------------|--------------------------|--------------------------|--------------------------|
| Baseline stability? | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| Resolution? | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

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	YES	NO	N/A
Peak shape?	<input type="checkbox"/>	___	___
Full-scale graph (attenuation)?	<input type="checkbox"/>	___	___
Other:_____	<input type="checkbox"/>	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?	<input type="checkbox"/>	___	___
ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, reject all positive data.			
10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	<input type="checkbox"/>	___	___
10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?	<input type="checkbox"/>	___	___
10.7 Do the relative intensities of the characteristic ions in the sample agree within $\pm 30\%$ of the corresponding relative intensities in the reference spectrum?	<input type="checkbox"/>	___	___
ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.			
ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any Positive compound identification.			
11.0 <u>Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)</u>			
NOTE: Use this section only if TIC are required.			
11.1 Are all Tentatively Identified Compound reporting forms present; and do listed TIC's include scan number or	<input type="checkbox"/>	___	___

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retention time, estimated concentration and a qualifier?

NOTE: Add "N" qualifier to all TIC's which have CAS number, if missing.

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

- a. Samples and/or fractions as appropriate ☐ ☐ ☐
- b. Blanks ☐ ☐ ☐

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS #.

NOTE: If TIC's are present in the associated blanks take action as specified in section 7.2 above.

11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)? ☐ ☐ ☐

ACTION: If yes, document in the data assessment that non VOA Compounds are present in the sample(s).

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? ☐ ☐ ☐

11.5 Do TIC and "best match" standard relative ion intensities agree within $\pm 20\%$? ☐ ☐ ☐

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂ (M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal ☐ ☐ ☐

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YES NO N/A

standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions? ☒ ☐ ☐

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration? ☒ ☐ ☐

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)

14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction? ☒ ☐ ☐

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

14.2 Are all average RRFs > 0.050? ☒ ☐ ☐

ACTION: Circle all outliers with red pencil.

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ACTION: For any target analyte with average RRF < 0.05, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration. The % relative standard deviation (%RSD) \leq 20.0% as per Method 524.2-20, Sect. 10.2.6.1. ☐ ☐ ☐

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF? ☐ ☐ ☐

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) ☐ ☐ ☐

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)

15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest? ☐ ☐ ☐

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? ☐ ☐ ☐

NOTE: The mean response factors calculated during initial calibration are used for sample quantitation (Method 524.2-26, Sect. 12.1.1).

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YES NO N/A

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification ☐ ☐ ☐
determined using RRF and by CF?

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) ☐ ☐ ☐
between the initial and continuing RRF or CF which exceeds
30% (Method 524.2-21, Sect. 10.3.5).

ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

15.5 Do any volatile compounds have a RRF < 0.05? ☐ ☐ ☐

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, qualify all positive results for That analyte "J" and all non-detect results for that analyte "R".

15.6 Are there any transcription/calculation errors in the ☐ ☐ ☐
reporting of %D between initial and continuing RRF's/
CF's? (Check at least two values but if errors are found,
check more).

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

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YES NO N/A

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard areas on the internal standard ☐ ☐ ☐ reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid point calibration and (-30% to +100%) of the corresponding continuing calibration check (Method 524.2-21, Sect. 10.3.4)? The upper limits for internal standard areas have not been defined in the method. See action On the next page.

ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

- ACTION:
1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.
 2. Do not qualify non-detects when the associated IS Area is above the upper limit (+ 100%).
 3. If the IS area is below the lower limit (- 50% for initial calibration and -30% for the corresponding continuing calibration), qualify all associated non-detects "UJ".
 4. If extremely low area counts are reported (< 25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within ☐ ☐ ☐ 3 standard deviations of the mean retention compounds in the associated initial mid-point calibration standard Method 524.2-25, Sect.11.6)?

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YES NO N/A

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 3 standard deviations.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis? ☐ ☐ ☐

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

STANDARD OPERATING PROCEDURE

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Method 524.2 (Rev. 4.2, 1995)

Date: October 2001
SOP HW-29, Rev. 1

YES NO N/A

DEFINITIONS**Acronyms:**

BFB	-	bromofluorobenzene
BNA	-	base neutral acid
CCC	-	calibration check compound
CF	-	calibration factor (without internal standards)
CLP	-	contract laboratory program
CRQL	-	contract required quantitation limit
% D	-	percent difference or percent drift
GC/MS	-	gas chromatography/mass spectroscopy
IS	-	internal standard
l	-	liter
LFB	-	laboratory fortified blank
LRB	-	laboratory reagent blank
LFM	-	laboratory fortified matrix
FRB	-	field reagent blank
Kg	-	kilograms
m	-	meter
mm	-	millimeter
m/z	-	mass to charge ratio
QC	-	quality control
RIC	-	reconstructed ion chromatogram
RPD	-	relative percent difference
RRF	-	relative response factor (requires internal standard)
RRT	-	relative retention time
RSD	-	relative standard deviation
RT	-	retention time
SDG	-	sample delivery group
SOP	-	standard operating procedure
SPCC	-	system performance check compound
TIC	-	tentatively identified compound
TCLP	-	toxicity characteristic leach procedure
ug	-	micrograms
VOA	-	volatile organic acid

STANDARD OPERATING PROCEDURE

US EPA Region II
Method 524.2 (Rev. 4.2, 1995)

Date: October 2001
SOP HW-29, Rev. 1

YES NO N/A

DEFINITIONS

Data Qualified Definitions:

- U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
- NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.